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Office of the Principal, Government College, Ajmer

No. 1985 Date 9/5/2016.

To,

The Director (Research)

MaharshiDayanandSaraswati University,

Ajmer- 305001 (Rajasthan)

Sub - Synopsis for registration of Ph.D. degree in chemistry

Sir,

Enclosed please find herewith four copies of synopsis entitled **"Synthesis, Characterization and Antibacterial Activity of O-alkyl /O- aryl trithiophosphatesof Gp-5 (V, Nb, Ta) And Gp-7 (Mn, Tc, Re) Elements"**of **SumanBhatti** for the registration of Ph.D. degree (Chemistry) under supervision of **Dr. AlokChaturvedi** in the Subject of chemistry.

Kindly acknowledge the same.

Yours sincerely

Principal
S.P.C. Govt. College, Ajmer

Principal
Samrat Prithviraj Chauhan
Govt. College, Ajmer

Enclosed - Four copies of synopsis.

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To,

The Director (Research)

MaharshiDayanandSaraswati University,

Ajmer – 305001 (Rajasthan)

Through – Principal, S.P.C. Government College, Ajmer

Sub – Submission of synopsis in chemistry.

Sir,

I am submitting four copies of synopsis entitled **"Synthesis, Characterization And Antibacterial Activity of O-alkyl /O- aryl trithiophosphates of Gp-5 (V, Nb, Ta) And Gp-7 (Mn, Tc, Re) Elements"**for registration of Ph.D. in chemistry under supervision of **Dr. Alok Chaturvedi**, Lecturer, S.P.C. Government College, Ajmer.

Yours sincerely

SBhatti

(SumanBhatti)

Research Scholar

Lecturer

Dr. AlokChaturvedi

Govt. College, Ajmer

Supervisor

Lecturer (Selection Scale)

Department of Chemistry

S.P.C. Govt. College,Ajmer.

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SYNTHESIS, CHARACTERIZATION AND ANTIBACTERIAL ACTIVITY OF O-ALKYL / O-ARYL TRITHIOPHOSPHATES OF Gp-5(V,Nb,Ta) AND Gp-7(Mn,Tc,Re)ELEMENTS

समूह–5(V,Nb,Ta)तथा समूह–7(Mn,Tc,Re) के तत्वों का संश्लेषण, अभिलक्षण, प्रतिजीवाणुक सक्रियता

A

SYNOPSIS

Submitted for the Award of Ph.D. degree of

MaharshiDayanandSaraswati University, Ajmer

In the Faculty of Science (Chemistry)

By

SumanBhatti

Under Supervision of

Dr. AlokChaturvedi

Lecturer (Selection scale)

Department Of Chemistry

S.P.C. Govt. College, Ajmer.

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APPENDIX – IV

Format for outline of Proposed Research Work

(To be supplied in Eight copies)

1. Name of scholar

(In English) : **Suman Bhatti**

(In Hindi) : सुमन भट्ठी

2. Title of Research Work

(In English) :**Synthesis Characterization And Antibacterial Activity of O-alkyl /O- aryl trithiophosphatesof Gp-5 (V, Nb, Ta) and Gp-7 (Mn, Tc, Re) Elements**

(In Hindi) :**समूह–5 (V, Nb, Ta)तथा समूह–7 (Mn, Tc, Re) के तत्वों का संश्लेषण, अभिलक्षण, प्रतिजीवाणुक सक्रियता**

3. Location : **DEPARTMENT OF CHEMISTRY, S.P.C. GOVT. COLLEGE, AJMER**

4. Importance of proposed investigation, objectives : **Annexure – I**

5. Review of work already done on the subject of proposed research study : **Annexure – II**

6. Research gaps identified in the proposed field of investigation : **Annexure – III**

7. Major hypothesis, if any : **Annexure – IV**

8. Research methodology : **Annexure - V**

9. Bibliography : **Annexure - VI**

Lecturer Samir Prithviraj Chauhan Govt. College, Ajmer

Name & Signature of Supervisor with date & seal

Bhatti

Signature of Candidate with date 6/5/2016

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Annexure-I

IMPORTANCE OF PROPOSED INVESTIGATION

The organic derivatives of transition metal with sulphur and phosphorus containing ligands have important place in metallo organic chemistry.The synthesis and spectral aspects of partial esters of phosphoric acid and thiophosphoric acid of metallic and organometallic compound have been recently reviewed[1-6]. The interest in the chemistry of derivatives arises from their utility as pesticides[7],insecticides[8],bactereocides[9] and lubricant oil[10]etc.

Aliphatic monothiophosphates, dithiophosphates and trithiophosphates are used in defoliation, improvement in crop value and indirect control of pest invfestation.Organic trithiophosphate esters have been used as defoliants[11],insecticides[12], nematodicides and inhibitor in steel corrosion[13].

Due to unexceptional bonding modes (monodentate,bidentate,chelating or bridging) of metal complexes with sulphur and phosphorus containing ligands , and a wide range of biological and industrial applications , their interpretation possess challenges.

Due to interesting structural features and wide range of applications of the ligands containing both sulphur and phosphorus ,it was thought worthwhile to synthesize new complexes of O-alkyl or O-aryl trithiophosphate of gp-5(V,Ta,Nb) and gp-7(Mn,Tc,Re).

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OBJECTIVES

Objectives of proposed study are as follow:

1. To synthesize O-alkyl /O-aryl trithiophosphate ligand.

2. To synthesize complexes of O-alkyl andO-aryl trithiophosphates of gp-5(V,Nb,Ta) and gp-7(Mn,Tc,Re)

3. To characterize newly synthesize complexes by physico-chemical techniques viz: elemental analaysis ,molecular weight measurements, electrical conductivies.

4. To analyse structure of newly synthesize complexes with the help of spectroscopic techniques (IR,^{1}H,^{13}C,^{31}P NMR).

5. To detect newly synthesize compounds as antibacterical agent by paper disc method.

6. To compare antibacterial activity of derivatives with standard drugs.

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ANNXEURE-II

REVIEW OF WORK ALREADY DONE ON THE SUBJECT OF PROPOSED RESEARCH STUDY

A large number of metal derivatives of dithiophosphate ligands have been synthesized in recent years due to their antibacterial activity as well as interesting structural features[14-22].

A few metal complexes of O-alkyl/O-aryl trithiophosphates have also been synthesized and characterized by researchers in our laboratory as well as others[23-34].

Synthesis of triorganotin (IV) O- alkyl/O-phenyl trithiophosphates[23] have been carried out by the reaction of triorganotin(IV) chloride with dipotassium salt of O- alkyl/O-phenyl trithiophosphates in molar ratio 2:1 in anhydrous benzene.

$$2R_3SnCl + R'OPS_3K_2 \longrightarrow [(RO)P(S)(SSnR'_3)_2] + 2KCl\downarrow$$

($R=Pr^i, Bu^i, Ph$; $R'=Me, Pr^i, Bu, Ph$)

Reaction of antimony (V) bromide carried out with dipotassiumO-alkyl trithiophosphates in 1:1 molar ratio in methanol to synthesize triphenylantimony(V)-(O-alkyl, O-cycloalkyl and O-aryl) trithiophosphates[24]. Trigonalbipyramidal (TBP) geometry has been proposed to the newly synthesized compounds.

Diisopropyltin(IV) trithiophosphates have been synthesized[25] by the reaction of diisopropyltin dichloride with dipotassium salt of O-alkyl trithiophosphates in 1:1 molar ratio in methanol.

$$Pr^i_2SnCl_2 + (RO)P(S)(SK_2) \xrightarrow{CH_3OH} Pr^i_2Sn[S_2(S)P(OR)] + 2KCl$$

Complexes of bismuth (III) (O-alkyl, O-cycloalkyl and O-aryl) trithiophosphates, like $[(RO)P(S)S_2]_3 Bi_2$[26](R=Me, Et, Pr^i, Bu^n, Bu^s, Bu^i, Ph and cyclophenyl) synthesized by reaction of bismuthchloride with potassium trithiophosphate in 2:3 molar ratio. Reactivity of synthesized compounds studied by the reactions with halogens and lewis acids (such as anhydrous $AlCl_3$) .

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1:1:1 molar ratio of diorganotinchlorideR_2SnCl_2(R = Me,Bu,Ph) ,acetylacetone and O-alkyl trithiophosphates $R'OPS_3K_2$ (R'=Me, Et, Pr^i, Bu^i, Ph) refluxed in benzene . As a result compounds of type $R_2Sn[SSH(S)POR'][MeCOCHCOMe]$ have been synthesized[27]. Newly synthesized compounds found to have antifungal activity .The products are light yellow colored crystalline solids ,soluble in organic solvents but highly sensitive towards atmospheric moisture.

Reaction of tin tetrachloride with dipotassium salt of O-alkyltrrithiophosphate in methanol in 1:2 molar ratio yield bis-(O-alkyltrithiophosphato)tin(IV)[28] , $[ROP(S)S_2]_2Sn$ and their adducts $[(RO)P(S)S_2]_2$ and N-donar bases in 1:1 molar ratio. Four and six coordination suggested for bis-O-alkyltrithiophosphato)tin and their adducts with N-donar bases,respectively.

Complexes of type $R_2Sn[PhCOCHCOMe][SSH(S)POR']$ were synthesized and characterized by spectral studies (IR,^1H, ^{13}C, ^{31}P, ^{119}Sn NMR). Antibacterial activity of complexes were tested against gram positive and gram negative bacteria[29].

p-nitrophenyl and p-nitrobenzoyl derivatives of O,O'alkylenedithiophosphate have been synthesized[30].In contrast to the bidentate chelating behaviour of the ligand in the metallic and organometallic derivatives of alkylene dithiophosphates,the behaviour of dithiophosphato moiety in these derivatives is found to be monodentate in nature.

A series of mixed ligand complexes have been synthesized[31]. Reaction of diorganitin(IV) dichloride with dibenzoyl methane and O-alkyltrithiophosphates in 1:1:1 molar ratio yied products of type $[PhCOCHCOPh]R_2Sn[SSH(S)POR']$. Complexes werw found to have antibacterial activity.

Titanium tetrachloride reacts with potassium trithiophosphatess in 1:1 molar ratio in anhydrous benzene to synthesize dichlorotitanium(IV) trithiophosphates of type $TiCl_2[ROP(S)S_2]$[32] (where R=Me, Et, Pr^i, Bu^i, Ph and cyclohexyl). When these compounds studied in dry benzene by passing H_2S gas, sol-gel chemistry has been found.

Reaction of arsenic trichloride with dipotassium salt of O-alkyl / O-aryltrithiophosphate carried out in 1:1 and 2:3 molar ratio for the synthesis of arsenic (III) O-alkyl / O-aryl trithiophosphate of type $ClAs[S_2(S)P(OR)]$ and $ROP(S)[SAsS_2P(S)OR]_2$[33],respectively. Elemental analysis, molecular weight measurement and spectroscopic studies (IR, ^1H, and ^{31}P-NMR) used to characterize the newly synthesized complexes . Complexes have distorted tetrahedral geometry and good antibacterial activity.

Substituted organic esters (RO) $PS_3(C_6H_4CH_2)_2$ and $ROPS_3(RCO)_2$ synthesized[34] by the reaction of dipotassium salt of O-alkyl trithiophosphate with acetyl, benzyl and benzoyl chloride. These compounds are white crystalline solids and soluble in DMSO. These complexes have distorted tetrahedral geometry.

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ANNXEURE-III

RESEARCH GAPS IDENTIFIED IN PROPOSED FIELD OF INVESTIGTION

Review of literature reveals thatin the last few years , considerable work have been done in the field of metal derivatives of monothiophosphate and di-thiophosphate ligands .

Although few metal derivatives of O-alkyl trithiophosphates are investigated in our as well as some other laboratories .But no work has been done on metallic and organometallic derivatives of O-alkyl/ O-aryl trithiophosphates of gp-5(V, Nb, Ta) and gp-7(Mn, Tc, Re).

In the view of this, it was worthwhile to synthesize O-alkyl/ O-aryl trithiophosphates of gp-5(V, Nb, Ta) and gp-7(Mn, Tc, Re) and study their bonding modes,antibacterial activity and compare them with standard drugs.

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ANNXEURE-IV

MAJOR HYPOTHESIS, IF ANY

Metallic or/and organometallic derivatives of O-alkyl or O-aryl trithiophosphates will be synthesized by following procedure –

1. **Preparation of ligand-**
   A mixture of triethylamine and appropriate alcohol will be added dropwise to the suspension of P_2S_5 in benzene, results in formation of two layers. Two layers will be separated and methanolicsolution of 1mol of KOH will be added to the lower layer. The reaction will be shown as follow:

   $$P_2S_5+Et_3N + ROH \xrightarrow{KOH} K_2S_3POR.$$

   [R=Me, Et, Pr^i, Bu^i, Ph]

2. **Reaction of ligand with metallic and organometallic moieties –**
   Metallic and organometallic derivatives of O-alkyl /O-aryl trithiophosphateswill be prepared by treating potassium salt of O- alkyl /O-aryl trithiophosphates with metallic or/and organometallic halides of gp-5(V, Nb, Ta) and gp-7(Mn, Tc, Re) elements.

   $$MX_3/MX_5+K_2S_3POR \longrightarrow XMS_3POR / X(MS_3POR)_2 +KCl$$

   (Where M = V, Nb, Ta, Mn, Tc, Re)
   (R =Me, Et, Pr^i, Bu^i, Ph)

   Characterization of newly synthesized derivatives will be carried by elemental analysis, molecular weight measurement and spectroscopic methods (IR, ^1H, ^{13}C, ^{31}P –NMR).

   Antibacterial activity of synthesized complexes will be screen by using Paper disc method. Their antibacterial activity will be screen against E.coli, Entercocci and Klebsiella using Muller Hinten Agar media (HI media). Impinenem and Linezolid will be used as standard drugs.

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Annexure-V

RESEARCH METHODOLOGY

1. O-alkyl/O-aryl trithiophosphates will be synthesized by reaction of phosphorus pentasulphide with appropriate alcohol and triethylamine followed by reaction with potassium hydroxide in methanol.

2. Methanol, ethanol, isopropanol ,isobutanol, phenol, benzyl alcohol will be used in synthesis of potassium salt of O-alkyl/O-aryl trithiophosphates.

3. Metal derivatives of gp-5(V,Ta, Nb) and gp-7 (Mn, Tc, Re) with O-alkyl/O-aryl trithiophosphates will be synthesized by the reaction of metallic and organometallic halide with dipotassium salt of O-alkyl/O-aryl trithiophosphates.

4. Elemental analysis,molecular weight measurement and spectroscopic studies (IR, ^{1}H, ^{13}C, ^{31}P-NMR) will be recorded to characterize the complexes . Structure and co-ordination behavior will be assigned by using variation in absorbtion band.

5. Antibacterial and antifungal activity will be evaluated by using paper disc method and radial growth method, respectively. Results will be compared with standard drugs.

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Chapter wise Details of Proposed Research Work

Synthesis, Characterization And Antibacterial Activity of O-alkyl /O- aryl trithiophosphates of Gp-5 (V, Nb, Ta) And Gp-7 (Mn, Tc, Re) Elements

Chapter 1 - Introduction

Chapter 2 - Review of literature

Chapter 3 - Experimental (Methods and Materials)

Chapter 4 - Result and discussion

Chapter 5 - Summary

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CERTIFICATE BY SUPERVISOR

Certified that:-

1. I am willing to guide the research work of **Mrs. Suman Bhatti** on the above subject if she registered for Ph.D.
2. To the best of my knowledge the subject selected has not been studied so far in any university.
3. The subject is of sufficient scope to keep the candidate engaged for two years.
4. The subject will lead to a valuable contribution. I have seen and approved the outline and bibliography, etc. submitted by the candidate.
5. Necessary facilities are available in the department for the above research work (Certificate from Principal/Head University Department with regards to facilities available is enclosed).
6. I have Nil.. research candidate registered under my supervision and the serial no. of this candidate whose application being forwarded for registration is ..02..
7. The candidate is not a close relation (the term close relation is defined in examination ordinance of the university).

Signature of the Supervisor
with date

Lecturer.....
Samrat Prithviraj Chauhan
Govt. College, Ajmer

Certified that the available facilities will be extended to Mrs. Suman Bhatti for research work if she registered for Ph.D.

Signature of Principal/
Director of the Institute
with date

Principal
Samrat Prithviraj Chauhan
Govt. College, Ajmer

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